

## English Translation

## PATENT ABSTRACTS OF JAPAN

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(54) SILICONE RUBBER SPONGE FORMING COMPOSITION FOR FIXING ROLLER, FIXING ROLLER, AND METHOD FOR MANUFACTURING THE SAME

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a silicone rubber sponge forming composition for a fixing roller, which can be made as a cured product forming a fine and uniform cell, having low thermal conductivity, and superior in durability, and to provide the fixing roller, made of the same, having the low thermal conductivity, and superior in the durability.

SOLUTION: The silicone rubber sponge forming composition for the fixing roller contains (A) 100 pts.mass of a liquid organopolysiloxane including at least two alkenyl groups combined with silicon atoms in one molecule, (B) 0.1-30 pts.mass of a liquid organohydrogenpolysiloxane including at least two hydrogen atoms combined with silicon atoms in one molecule, (C) 1-20 pts.mass of a surfactant, (D) catalytic amount of an addition reaction catalyst, (E) 0-10 pts.mass of reinforcing silica fine powder, and (F) 30-200 pts.mass of water. The fixing roller formed using the composition and the method for manufacturing the same are provided.

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## CLAIMS

[Claim(s)]

[Claim 1]

- (A) Liquefied organopolysiloxane 100 mass part which contains in a monad an alkenyl group combined with at least two silicon atoms  
 (B) 0.1 to liquefied ORGANO hydrogen polysiloxane 30 mass part which contains in a monad a hydrogen atom combined with at least two silicon atoms  
 (C) One to surface-active agent 20 mass part  
 (D) An addition reaction catalyst A catalyst amount  
 (E) Zero to reinforcement nature silica impalpable powder 10 mass part  
 (F) 30 to water 200 mass part

A silicone rubber sponge plasticity constituent for fixing rollers to contain.

[Claim 2]

- (A) The silicone rubber sponge plasticity constituent for fixing rollers according to claim 1 whose mole ratio (a Si-H group / alkenyl group) of an alkenyl group of an ingredient and a silicon atom absorbed water matter atom of the (B) ingredient is 2.0 or less.

[Claim 3]

- (A) The silicone rubber sponge plasticity constituent for fixing rollers according to claim 1 or 2 whose degree of polymerization of organopolysiloxane of an ingredient is 400 or less.

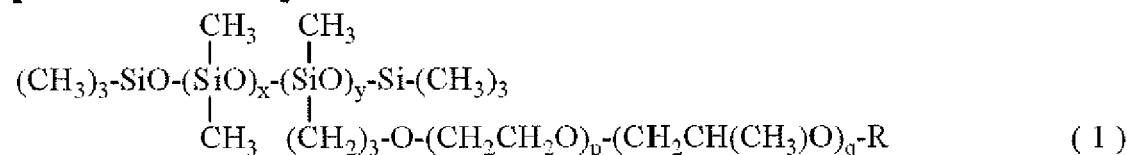
[Claim 4]

- (C) The silicone rubber sponge plasticity constituent for fixing rollers according to claim 1, 2, or 3 whose surface-active agent of an ingredient is polyether denaturation silicone.

[Claim 5]

The silicone rubber sponge plasticity constituent for fixing rollers according to claim 4 whose polyether denaturation silicone is what is expressed with a following formula (1).

[Chemical formula 1]



R is H or CH<sub>3</sub> among [type, and 10-200y of x are 1-20, and x/y is an integer with which it is satisfied of 5-50, and 3-50q of p are 0-30, and p/(p+q) is an integer with which it is satisfied of 0.5-1. Molecular weights are 2,000-50,000 and the amount of siloxanes in a molecule is 40 to 95 mass %.]

[Claim 6]

A silicone rubber sponge plasticity constituent for fixing rollers of Claims 1-5 whose thermal

conductivity of a hardened material is below 0.15 W/m and \*\* given in any 1 clause.

[Claim 7]

It is a heat fusing roller with which it comes to form a silicone rubber sponge layer in a peripheral face of roll axes, A silicone rubber sponge fixing roller with which silicone rubber sponge which forms this silicone rubber sponge layer is characterized by making it come to harden a silicone rubber sponge plasticity constituent of Claims 1-6 given in any 1 clause.

[Claim 8]

A manufacturing method of a silicone rubber sponge fixing roller which makes a silicone rubber sponge layer form in it by volatilizing moisture at temperature of not less than 100 \*\* after making a peripheral face of roll axes harden a silicone rubber sponge plasticity constituent of Claims 1-6 given in any 1 clause at less than 100 \*\*.

[Claim 9]

It is a fluororesin coating fixing roller with which it comes to form a fluoro-resin or fluororubber layers in a peripheral face of roll axes via a silicone rubber sponge layer, A fluoro-resin or a fluorocarbon rubber covering silicone rubber sponge fixing roller with which silicone rubber sponge which forms this silicone rubber sponge layer is characterized by making it come to harden a silicone rubber sponge plasticity constituent of Claims 1-6 given in any 1 clause.

[Claim 10]

A peripheral face of roll axes is made to harden a silicone rubber sponge plasticity constituent of Claims 1-6 given in any 1 clause at less than 100 \*\*, A manufacturing method of a fluoro-resin which grinds the silicone rubber sponge layer surface and makes a fluoro-resin or fluororubber layers form in this surface after making a silicone rubber sponge layer form by volatilizing moisture at temperature of not less than 100 \*\*, or a fluorocarbon rubber covering silicone rubber sponge fixing roller.

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[Translation done.]

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## DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[Field of the Invention]

[0001]

This invention relates to a silicone rubber sponge plasticity constituent for fixing rollers of the heat fixing equipment in electrostatic recording devices, such as an electrophotography copying machine, a printer, and a facsimile, a fixing roller formed using this constituent, and a manufacturing method for the same.

[Background of the Invention]

[0002]

A heat cure type liquid silicone rubber composition is excellent in a moldability, and after shaping is used in various fields from excelling in heat resistance and electric insulation. In it, since it excels in heat resistance or a mold-release characteristic, it is used for fixing rollers, such as PPC, LBP, and FAX. In the apparatus using these electrophotography processes, it is necessary to fix to tracing paper the toner image transferred by tracing paper from the photo conductor surface. As a method of fixing this toner image, tracing paper is passed between the heater roll and pressure roll which are carrying out contact rotation mutually and which were heated, thermal melting arrival carries out the toner image on tracing paper, and the method of fixing is adopted widely. Although it can be considered as the quick copying machine of a response, a printer, etc. by generally making the thermal conductivity of a roll material high in this thermal melting arrival method, On the other hand, as for the thermally conductive high thing, as for heat dissipation, a good material of heat storage capability is early needed conversely low [ thermal conductivity ] in the flow of a miniaturization and low-pricing. There is silicone rubber foam which used gaseous low-fever conductivity as this material, and there are a method of using the hydrogen gas which carries out a byproduction as a process of this foam at the time of the method of adding a heat decomposition type blowing agent or hardening, etc.

[0003]

However, the method of adding a heat decomposition type blowing agent lets the toxicity of the cracked gas, and a smell be problems.

Hardening inhibition by a foaming agent was made into the problem in what uses a platinum catalyst for a curing catalyst.

In the method of using the hydrogen gas which carries out a byproduction at the time of hardening, there were problems, such as requiring cautions for the handling at the time of the explosivity of hydrogen gas and preservation of uncured material. In shaping made to foam within a metallic mold like injection moulding, there was a problem that it was difficult to obtain the silicone rubber foam which has a minute and uniform cell.

[0004]

Although the method of blending a hollow filler was shown by JP,2000-143986,A (patent documents

1), there was a problem that a hollow filler was dramatically lightweight and combination was difficult. By JP,H6-207038,A (patent documents 2), after adding an emulsifier and water to the addition hardening setup-of-tooling product of silicone and making it distribute water, the method of volatilizing water and obtaining sponge is indicated, but about a fixing roller use, it is not suggested at all. Although silica impalpable powder etc. are used as the essential ingredient as a viscous agent, it is not preferred as an object for fixing rollers to blend these so much, in order to worsen a compression set.

[0005]

[Patent documents 1] JP,2000-143986,A

[Patent documents 2] JP,H6-207038,A

[Description of the Invention]

[Problem to be solved by the invention]

[0006]

This invention was made in view of the above-mentioned situation, and forms a detailed and uniform cell, and an object of this invention is to provide a silicone rubber sponge plasticity constituent for fixing rollers which gives the hardened material which is low-feeve conduction and was excellent in endurance, a fixing roller formed using this constituent, and a manufacturing method for the same.

[Means for solving problem]

[0007]

this invention person found out that the obtained silicone rubber sponge was suitable as a fixing roller use by blending a surface-active agent and water with an addition hardening type silicone rubber composition, as a result of inquiring wholeheartedly, in order to attain the above-mentioned purpose.

Namely, the liquefied organopolysiloxane which contains the alkenyl group combined with at least two silicon atoms in the (A) monad, (B) The liquefied ORGANO hydrogen polysiloxane which contains in a monad the hydrogen atom combined with at least two silicon atoms, (C) By using a surface-active agent and the silicone rubber sponge plasticity constituent for fixing rollers which contains the specific amount of polyether denaturation silicone, the (D) addition reaction catalyst, (E) reinforcement nature silica impalpable powder, and (F) water preferably, A detailed and uniform cell is formed, and it finds out that the fixing roller which is low-feeve conduction and was excellent in endurance can be manufactured, and came to make this invention.

[0008]

Therefore, this invention provides a silicone rubber sponge plasticity constituent for the following fixing rollers, a fixing roller, and a manufacturing method for the same.

[1] Liquefied organopolysiloxane 100 mass part which contains the alkenyl group combined with at least two silicon atoms in the (A) monad

(B) 0.1 to liquefied ORGANO hydrogen polysiloxane 30 mass part which contains in a monad the hydrogen atom combined with at least two silicon atoms

(C) One to surface-active agent 20 mass part

(D) Addition reaction catalyst Catalyst amount

(E) Zero to reinforcement nature silica impalpable powder 10 mass part

(F) 30 to water 200 mass part

The silicone rubber sponge plasticity constituent for fixing rollers to contain.

[2] It is a heat fusing roller with which it comes to form a silicone rubber sponge layer in the peripheral face of roll axes, The silicone rubber sponge fixing roller with which the silicone rubber sponge which forms this silicone rubber sponge layer is characterized by making it come to harden the above-mentioned silicone rubber sponge plasticity constituent.

[3] A manufacturing method of a silicone rubber sponge fixing roller which makes a silicone rubber sponge layer form in it by volatilizing moisture at temperature of not less than 100 °C after making a peripheral face of roll axes harden the above-mentioned silicone rubber sponge plasticity

constituent at less than 100 \*\*.

[4] It is a fluoro-resin coating fixing roller with which it comes to form a fluoro-resin or fluororubber layers in a peripheral face of roll axes via a silicone rubber sponge layer, A fluoro-resin or a fluorocarbon rubber covering silicone rubber sponge fixing roller with which silicone rubber sponge which forms this silicone rubber sponge layer is characterized by making it come to harden the above-mentioned silicone rubber sponge plasticity constituent.

[5] A peripheral face of roll axes is made to harden the above-mentioned silicone rubber sponge plasticity constituent at less than 100 \*\*, A manufacturing method of a fluoro-resin which grinds the silicone rubber sponge layer surface and makes a fluoro-resin or fluororubber layers form in this surface after making a silicone rubber sponge layer form by volatilizing moisture at temperature of not less than 100 \*\*, or a fluorocarbon rubber covering silicone rubber sponge fixing roller.

[Effect of the Invention]

[0009]

The fixing roller which formed the cell detailed [ the silicone rubber sponge plasticity constituent for fixing rollers of this invention ] and uniform, thermal conductivity was low, could become a hardened material which is excellent in endurance, and was formed using this is low-feeve conduction, and is excellent in endurance.

[Best Mode of Carrying Out the Invention]

[0010]

If this invention is explained in more detail, the silicone rubber sponge plasticity constituent for fixing rollers of this invention contains following (A) - (F) ingredient.

(A) Liquefied organopolysiloxane 100 mass part which contains in a monad the alkenyl group combined with at least two silicon atoms

(B) 0.1 to liquefied ORGANO hydrogen polysiloxane 30 mass part which contains in a monad the hydrogen atom combined with at least two silicon atoms

(C) One to surface-active agent 20 mass part

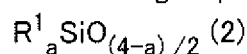
(D) Addition reaction catalyst Catalyst amount

(E) Zero to reinforcement nature silica impalpable powder 10 mass part

(F) 30 to water 200 mass part

[0011]

(A) The liquefied organopolysiloxane which contains the alkenyl group combined with at least two silicon atoms in the monad of an ingredient, It is base resin (base polymer) of this constituent, and what is shown with the following average composition formula (2) can be used as this alkenyl-group content organopolysiloxane.



[0012]

inside of formula, and  $R^1$  -- mutual -- the carbon numbers 1-10 same or of a different kind -- they are unsubstituted [ of 1-8 ], or a substitution monovalent hydrocarbon group preferably -- a -- 1.5-2.8 -- desirable -- 1.8-2.5 -- it is a positive number of the range of 1.95-2.02 more preferably. As a monovalent hydrocarbon group of unsubstituted [ which was combined with the silicon atom shown by the above-mentioned  $R^1$  here ], or substitution, A methyl group, an ethyl group, a propyl group, an isopropyl group, a butyl group, an isobutyl group, A tert-butyl group, a pentyl group, a neopentyl group, a hexyl group, a cyclohexyl group, Alkyl groups, such as an octyl group, a nonyl group, and a decyl group, a phenyl group, a tolyl group, Aryl groups, such as a xylyl group and a naphthyl group, benzyl, a phenylethyl group, Aralkyl groups, such as a phenylpropyl group, a vinyl group, an allyl group, a propenyl group, An isopropenyl group, a butenyl group, a hexenyl group, a cyclohexenyl group, A chloropropyl group, alkenyl groups, such as an octenyl group, etc. and what replaced some or all of the hydrogen atom of these bases by halogen atoms, such as fluoride, bromine, and chlorine, a cyano group, etc., for example, a chloromethyl group, a bromoethyl group, a triphloropropyl group, a

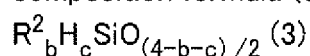
cianoethyl group, etc. are mentioned. In this case, at least two in  $R^1$  need to be an alkenyl group (the thing of the carbon numbers 2-8 is a thing of 2-6 desirable still more preferably.). As for the content of an alkenyl group, it is especially preferred that it is [ 0.01-10 mol ] % 0.001-20-mol% in  $R^1$ . It may combine with the silicon atom of molecular chain terminals, or may combine with the silicon atom in the middle of a chain, or this alkenyl group may be combined with both.

[0013]

The structures of this organopolysiloxane may be a branched state structure, cyclic structure, etc. selectively, although a main chain consists of a repetition of JIORUGANO siloxane units and chain both ends usually have straight-chain-shape structure on the basic target blocked by the Tori ORGANO siloxy group. If liquefied [ at a room temperature ] about a degree of polymerization (or the number of the silicon atoms in one molecule), it is good, but since water becomes difficult to distribute a hyperviscous thing, in a degree of polymerization, a degree of polymerization is [ a degree of polymerization ] usually 300 or less more preferably 400 or less 800 or less. Although the minimum is not limited, a degree of polymerization is [ degrees of polymerization ] usually 100 or more things preferably 50 or more.

[0014]

(B) The liquefied ORGANO hydrogen polysiloxane of an ingredient, Three or more (usually 3-200 pieces) things for which it has 3-100 silicon atom absorbed water matter atoms more preferably are preferably required in [ at least two ] a monad, and what is shown with the following average composition formula (3) can be used.



( $R^2$  is the substitution or the unsubstituted monovalent hydrocarbon group of the carbon numbers 1-10 among a formula.) 0.7-2.1c of b are 0.001-1.0, and b+c is a positive number with which it is satisfied of 0.8-3.0.

[0015]

Although what was illustrated by  $R^1$ , and the same thing can be mentioned as a monovalent hydrocarbon group of  $R^2$  here, what does not have an aliphatic unsaturated group is preferred. Preferably, it is desirable, 0.01-1.0, and b+c are preferred, 0.8-2.0c of b may be 1.0-2.5, and the molecular structure of the ORGANO hydrogen polysiloxane may be which structure of straight chain shape, annular, branched state, and three-dimensional mesh shape. In this case, as for the number of the silicon atoms in a monad (or degree of polymerization), 2-300 pieces and what is especially liquefied at the room temperature (25 \*\*) of about 4-150 pieces are used suitably. The hydrogen atom combined with a silicon atom may be located in any in the middle of molecular chain terminals and a chain, and may be located in both.

[0016]

As such an ORGANO hydrogen polysiloxane, Both-ends trimethylsiloxy group blockade methyl hydrogen polysiloxane, A both-ends trimethylsiloxy group blockade dimethylsiloxane methylhydrogensiloxane copolymer, Both-ends dimethyl hydrogen siloxy group blockade dimethylpolysiloxane, A both-ends dimethyl hydrogen siloxy group blockade dimethylsiloxane methylhydrogensiloxane copolymer, A both-ends trimethylsiloxy group blockade methylhydrogensiloxane diphenyl siloxane copolymer, A both-ends trimethylsiloxy group blockade methylhydrogensiloxane diphenyl siloxane dimethylsiloxane copolymer,  $(CH_3)_2$  The copolymer etc. which comprise the copolymer, and  $(CH_3)_2HSiO_{1/2}$  unit which comprises  $_2HSiO_{1/2}$  unit, and  $SiO_{4/2}$  unit,  $SiO_{4/2}$  unit, and  $SiO(C_6H_5)_3/2$  unit are mentioned.

[0017]

The loadings of this ORGANO hydrogen polysiloxane are 0.1 - 30 mass part to organopolysiloxane 100 mass part of the (A) ingredient, and are 0.3 - 20 mass part preferably. Less than 0.1 mass part

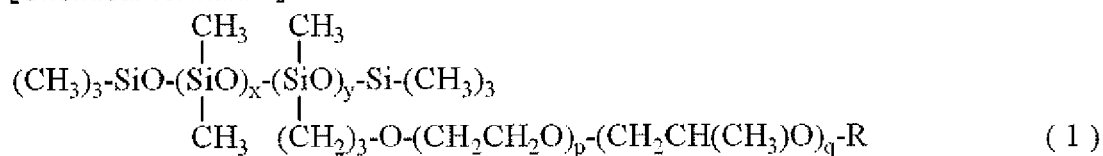
of hardening is [ loadings ] insufficient, they become gel, and a rubber-like elastic body hardened material cannot be given, but in the quantity exceeding 30 mass parts, the intensity and compression set-proof nature of a hardened material fall remarkably, and keep (a compression set becoming large).

The mole ratios (a Si-H group / alkenyl group) of the alkenyl group of the (A) ingredient and a silicon atom absorbed water matter atom are 0.5–1.6 especially preferably 2.0 or less preferably. When this ratio exceeds 2.0, a compression set not only becoming large but dehydrogenation has priority, and there is a possibility that a uniform detailed cell may no longer be obtained. When this ratio is too small, there is a possibility that rubber strength may become insufficient.

[0018]

(C) Any of an anionic system, a cation system, a dipolar ion system, and an Nonion system may be sufficient as the surface-active agent of an ingredient. As an anionic system surface-active agent, higher fatty acid salt, fatty alcohol sulfate, alkylbenzene sulfonates, alkylnaphthalenesulfonate, and polyethylene-glycol sulfuric ester salt can be mentioned, for example. As an Nonion system surface-active agent, for example Polyoxyethylene alkyl phenyl ether. Sorbitan fatty acid ester species, polyoxyethylene sorbitan fatty acid ester, polyoxyalkylene fatty acid ester, polyoxyethylene polyoxypropylene, and fatty acid monoglyceride can be mentioned. As a cation system surface-active agent, alkylamine salt, quarternary ammonium salt, and alkyl pyridinium salts can be mentioned, for example. The thing of carboxy betaine and a glycine type is mentioned as a dipolar ion system surface-active agent. It is desirable at the point that polyether denaturation silicone can form high durability sponge in a detailed cell in these. As such polyether denaturation silicone, what is expressed with a following formula (1) is preferred.

[Chemical formula 1]



[0019]

R is H or CH<sub>3</sub> among a formula -- x -- 10–200 -- it is 20–100 preferably -- y -- 1–20 -- it is 2–10 preferably and x/y is 5–50, and an integer with which it is preferably satisfied of 5–30. p -- 3–50 -- it is 3–40 preferably -- q -- 0–30 -- it is 0–20 preferably, and p/(p+q) is 0.5–1, and an integer with which it is preferably satisfied of 0.7–1.

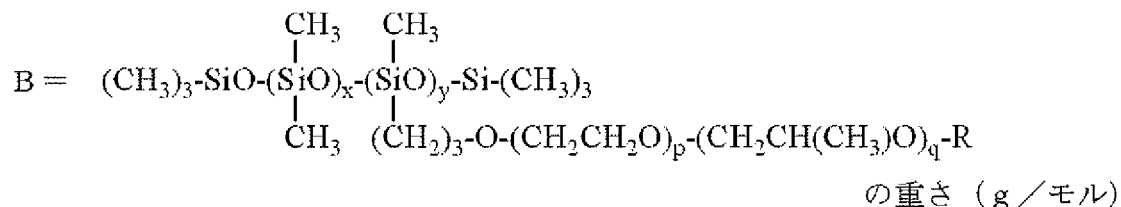
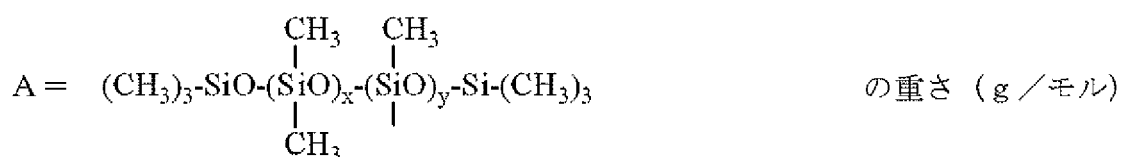
[0020]

a molecular weight -- 2,000–50,000 -- it is 2,000–20,000 preferably. In this case, this determination of molecular weight is a value by the weight average molecular weight of the polystyrene conversion by GPC (gel permeation chromatography). the amount of siloxanes in a molecule -- 40 to 95 mass % -- it is 50 to 85 mass % preferably. In this case, this amount of siloxanes is a value shown by x(A/B) 100 (mass %).

[0021]

[Chemical formula 2]





(However, R, x, y, p, and q are the same as that of the above.)

[0022]

These surface-active agents may be used alone, and two or more sorts may be used for them. Loadings of these surface-active agents are 2 – 15 mass part preferably one to 20 mass part to organopolysiloxane 100 mass part of the (A) ingredient. As for distribution of water, less than one mass part will be insufficient, and a detailed cell will not be obtained, but it will have an adverse effect on a rubber physical property in quantity exceeding 20 mass parts.

[0023]

(D) As an addition reaction catalyst of an ingredient, platinum system catalysts, such as a reactant of platinum black, the 2nd platinum of chloridation, chloroplatinic acid, chloroplatinic acid, and monohydric alcohol, a complex of chloroplatinic acid and olefins, and platinum screw acetoacetate, a palladium catalyst, a rhodium catalyst, etc. are mentioned. Loadings of this addition reaction catalyst can be made into a catalyst amount, and are usually about 1–500 ppm especially 0.5–1,000 ppm to the (A) ingredient as a platinum metal.

[0024]

(E) Although it is added in order to obtain rubber strength of a hardened material, and reinforcement nature silica impalpable powder of an ingredient is not limited in particular, fumed silica and precipitated silica are preferred for it. The surface treatment of such silica impalpable powder may be carried out, for example by chain organopolysiloxane, annular organopolysiloxane, hexamethyldisilazane, various organosilanes, etc.

As for specific surface area of reinforcement nature silica impalpable powder of this invention, it is preferred 50–350m<sup>2</sup>/g and that it is 80–250m<sup>2</sup>/g especially. Specific surface area can be determined, for example with a BET adsorption process.

[0025]

Ten or less (namely, zero to 10 mass part) mass parts of loadings [ eight or less mass part zero or more-mass part of ] of these reinforcement nature silica are five or less mass part zero or more-mass part more preferably to (A) ingredient 100 mass part. A compression set of a hardened material will worsen in quantity exceeding ten mass parts.

[0026]

(F) Loadings of water of an ingredient are 40 – 120 mass part preferably 30 to 200 mass part to (A) ingredient 100 mass part. As for a thermally conductive fall, less than 30 mass parts will be insufficient, and it will become difficult to make a detailed and uniform cell from quantity exceeding 200 mass parts.

[0027]

A bulking agent like [ if needed ] quartz powder, diatomaceous earth, and calcium carbonate as other ingredients, Resin of a silicone series used as a reinforcing agent, carbon black, a conductive

flower of zinc, Conducting agents, such as a metal powder, a nitrogen containing compound and an acetylene compound, phosphorus compounds, Hydrosilylation reaction controlling agents, such as a nitril compound, carboxylate, a tin compound, a mercury compound, and a sulfur compound, It is made arbitrary to blend internal release agents, such as iron oxide, a heat-resistant agent like cerium oxide, and dimethyl silicone oil, an adhesive grant agent, a thixotropic grant agent, etc. in the range which does not spoil the purpose of this invention.

[0028]

Although the fixing roller of this invention forms the high-heat-conductivity hardened material layer of the above-mentioned silicone rubber composition in rodding, the construction material of rodding, a size, etc. can be suitably selected in this case according to the kind of roll.

[0029]

Shaping of a silicone rubber composition and the hardening method can also be selected suitably, for example, it can fabricate by methods, such as injection molding, transfer molding, injection moulding, and coating, and hardens with heating. Under the present circumstances, less than 100 \*\* of temperature conditions [ not less than 80 \*\* less than 100 \*\* of / 80-98 \*\* of ] which stiffen rubber are usually 80-95 \*\* more preferably, and cure time is 2 hours from 15 minutes more preferably from 10 minutes from 5 minutes for 6 hours for 12 hours. If curing temperature will be not less than 100 \*\*, water will volatilize and desired sponge will no longer be obtained. As a secondary cure for volatilizing water, not less than 100 \*\* of temperature conditions [ not less than 150 \*\* 250 \*\* or less of ] are not less than 180 \*\* 250 \*\* or less more preferably, and secondary curing time is 24 hours from 30 minutes preferably from 10 minutes for 48 hours.

[0030]

A fluororesin layer and fluororubber layers may be further provided in the periphery of a silicon rubber layer. In this case, a fluororesin layer is formed by fluororesin coating material, a fluororesin tube, etc., and covers the above-mentioned silicon rubber layer. Here as a fluororesin coating material, For example, latex of polytetrafluoroethylene resin (PTFE), Die ERURA textile (the Daikin Industries, LTD. make, fluorine system latex) etc. are mentioned, and as a fluororesin tube, Can use a commercial item and For example, polytetrafluoroethylene resin (PTFE), Although tetrafluoroethylene perfluoroalkyl vinyl ether copolymer resin (PFA), fluoridation ethylene-polypropylene copolymer resin (FEP), polyvinylidene fluoride resin (PVDF), polyvinyl fluoride resin, etc. are mentioned, PFA is [ especially among these ] preferred.

[0031]

As for especially the hardened material layer of this silicone rubber sponge plasticity constituent, it is preferred that the thermal conductivity of below 0.15 W/m and \*\* is 0.05-0.14W/mand\*\*. If thermal conductivity is too small, a rubber physical property may be insufficient, and it may be inferior to roll endurance etc., or the performance as a high speed printer may become insufficient.

[0032]

Although the thickness of the above-mentioned silicon rubber layer is selected suitably, it is especially preferred that it is 0.1-50 mm 0.05-80 mm at the point of making use of the rubber elasticity of silicone rubber. As for especially the thickness of the fluoro-resin formed on it, or fluororubber layers, 10-100 micrometers is preferred 5-200 micrometers.

[Working example]

[0033]

Although an working example and a comparative example are shown and this invention is explained concretely hereafter, this invention is not restricted to the following working example. In the following example, a part and % show mass part and mass %, respectively. A molecular weight is the weight average molecular weight of the polystyrene conversion by GPC.

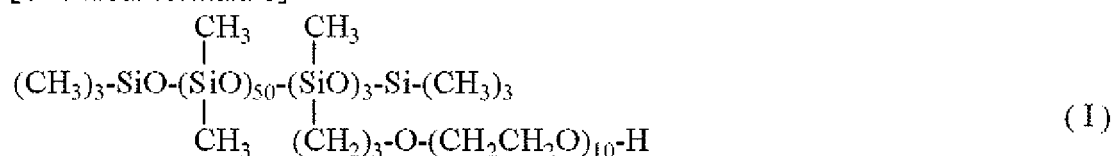
[0034]

[Working example 1]

100 copies of dimethylpolysiloxane (the degree of polymerization 220, vinyl value 0.000125 mol/g) by

which both ends were blocked by the dimethylvinyl siloxy group, three copies of fumed silica (the product made by Japanese Aerosil, R-972) whose specific surface area is 110m<sup>2</sup>/g and by which hydrophobing processing was carried out, following formula (I)

[Chemical formula 3]



Come out and eight copies of polyether denaturation silicone (the molecular weight 5540, 73% of the amount of siloxanes in a molecule) shown is put into a planetary mixer, After continuing churning for 10 minutes, 4.3 copies (silicon atom absorbed water matter atom / alkenyl-group (mole ratio) =1.3) of methyl hydrogen polysiloxane (the degree of polymerization 18, amount of Si-H 0.0038 mol/g) which has a Si-H basis in both ends and a side chain as a cross linking agent was added, and stirring was performed for 10 minutes. 80 copies of water was added in 4 steps to this, and stirring was continued for total 1 hour. Finally 0.05 copy of ethynylcyclohexanol was added as a reaction controlling agent, and a constituent done for churning continuously [ a constituent / for 15 minutes ] was used as a silicone rubber composition (1).

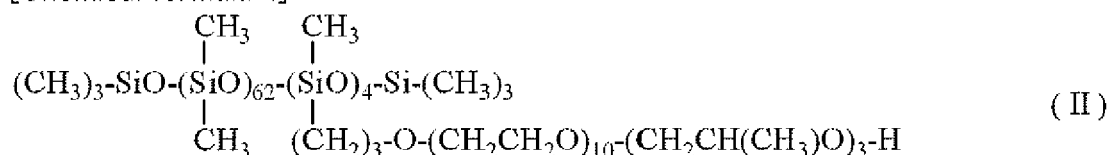
Mix 0.1 copy of platinum catalyst (1% of Pt concentration) to this silicone rubber composition (1), slush into a mold, and it is neglected in 95 \*\* oven for 1 hour, After making it harden, it removed from a mold and described in Table 1 about a result of having measured specific gravity, hardness, and a compression set based on JIS K 6249 about a hardened material obtained by carrying out postcure for 4 hours at 200 \*\*. Similarly it described in Table 1 about a result of having observed a cell state of a section with an electron microscope as a result of measuring thermal conductivity with a heat-conduction plan (QTM-3 Kyoto electronic company make).

[0035]

[Working example 2]

50 copies of side-chain vinyl group content dimethylpolysiloxane (the degree of polymerization 400, vinyl value 0.000094mol/g), 50 copies of dimethylpolysiloxane (degree of polymerization 180) by which both ends were blocked by the dimethylvinyl siloxy group, 0.5 copy of fumed silica (the product made by Japanese Aerosil, Aerosil 200) whose specific surface area is 190m<sup>2</sup>/g, following formula (II)

[Chemical formula 4]



Three copies of polyether denaturation silicone come out of and shown (the molecular weight 5460, 51% of the amount of siloxanes in a molecule), 3.2 copies (silicon atom absorbed water matter atom / alkenyl-group (mole ratio) =1.0) were put into the planetary mixer for methyl hydrogen polysiloxane of the working example 1 as a cross linking agent, and stirring was performed for 15 minutes. Then, 60 copies of water was added in 3 steps, and stirring was continued for total 1 hour. Finally, 0.05 copy of ethynylcyclohexanol was added as a reaction controlling agent, and the constituent done for churning continuously [ the constituent / for 15 minutes ] was used as the silicone rubber composition (2).

0.1 copy of platinum catalyst (1% of Pt concentration) was mixed to this silicone rubber composition

(2), like the working example 1, it was made to harden, specific gravity, hardness, a compression set, thermal conductivity, and the cell state of the section were measured, and it described in Table 1.

[0036]

[Working example 3]

50 copies of side-chain vinyl group content dimethylpolysiloxane (the degree of polymerization 400, vinyl value 0.000094mol/g), 50 copies of dimethylpolysiloxane by which both ends were blocked by the dimethylvinyl siloxy group (degree of polymerization 180), the fumed silica (the product made by Japanese Aerosil.) whose specific surface area is 190m<sup>2</sup>/g Aerosil 200 0.5 copy, three copies of polyether denaturation silicone shown by the above-mentioned formula (II), 7.0 copies (silicon atom absorbed water matter atom / alkenyl-group (mole ratio) =2.2) of methyl hydrogen polysiloxane of the working example 1 was put into the planetary mixer as a cross linking agent, and stirring was performed for 15 minutes. Then, 60 copies of water was added in 3 steps, and stirring was continued for total 1 hour. Finally, 0.05 copy of ethynylcyclohexanol was added as a reaction controlling agent, and the constituent done for churning continuously [ the constituent / for 15 minutes ] was used as the silicone rubber composition (3).

0.1 copy of platinum catalyst (1% of Pt concentration) was mixed to this silicone rubber composition (3), like the working example 1, it was made to harden, specific gravity, hardness, a compression set, thermal conductivity, and the cell state of the section were measured, and it described in Table 1.

[0037]

[Working example 4]

100 copies of dimethylpolysiloxane which both ends are blocked by a dimethylvinyl siloxy group, and contains a vinyl group in a side chain (the degree of polymerization 550, vinyl value 0.000120 mol/g), the fumed silica (the product made by Japanese Aerosil.) whose specific surface area is 110m<sup>2</sup>/g and by which hydrophobing processing was carried out R-972) Put eight copies of polyether denaturation silicone shown by three copies and the above-mentioned formula (I) into a planetary mixer, After continuing churning for 10 minutes, 4.1 copies (silicon atom absorbed water matter atom / alkenyl-group (mole ratio) =1.3) of methyl hydrogen polysiloxane of the working example 1 was added, and stirring was performed for 10 minutes. 80 copies of water was added in 4 steps to this, and stirring was continued for total 1 hour. Finally 0.05 copy of ethynylcyclohexanol was added as a reaction controlling agent, and the constituent done for churning continuously [ the constituent / for 15 minutes ] was used as the silicone rubber composition (4).

0.1 copy of platinum catalyst (1% of Pt concentration) was mixed to this silicone rubber composition (4), like an working example 1, it was made to harden, specific gravity, hardness, a compression set, thermal conductivity, and a cell state of a section were measured, and it described in Table 1.

[0038]

[An working example 5]

100 copies of dimethylpolysiloxane of an working example 1 by which both ends were blocked by a dimethylvinyl siloxy group (the degree of polymerization 220, vinyl value 0.0125mol/100 g), Three copies of fumed silica (a product made by Japanese Aerosil, R-972), following formula (III) whose specific surface area is 110m<sup>2</sup>/g and by which hydrophobing processing was carried out

[Chemical formula 5]



After having come out, putting eight copies of non-ion system surface-active agents shown into a planetary mixer and continuing churning for 10 minutes, 4.3 copies of methyl hydrogen polysiloxane of an working example 1 was added as a cross linking agent, and stirring was performed for 10 minutes. 80 copies of water was added in 4 steps to this, and stirring was continued for total 1 hour. Finally 0.05 copy of ethynylcyclohexanol was added as a reaction controlling agent, and a

constituent done for churning continuously [ a constituent / for 15 minutes ] was used as a silicone rubber composition (5).

0.1 copy of platinum catalyst (1% of Pt concentration) was mixed to this silicone rubber composition (5), like the working example 1, it was made to harden, specific gravity, hardness, a compression set, thermal conductivity, and the cell state of the section were measured, and it described in Table 1.

[0039]

[Comparative example 1]

100 copies of dimethylpolysiloxane by which the both ends of the working example 1 were blocked by the dimethylvinyl siloxy group (the degree of polymerization 220, vinyl value 0.0125mol/100 g), the fumed silica (the product made by Japanese Aerosil.) whose specific surface area is 110m<sup>2</sup>/g and by which hydrophobing processing was carried out R-972) After putting eight copies of polyether denaturation silicone shown by three copies and the above-mentioned formula (I) into the planetary mixer and continuing churning for 10 minutes, 4.3 copies of methyl hydrogen polysiloxane of the working example 1 was added as a cross linking agent, and stirring was performed for 10 minutes. Finally 0.05 copy of ethynylcyclohexanol was added as a reaction controlling agent, and the constituent done for churning continuously [ the constituent / for 15 minutes ] was used as the silicone rubber composition (6).

0.1 copy of platinum catalyst (1% of Pt concentration) was mixed to this silicone rubber composition (6), like the working example 1, it was made to harden, specific gravity, hardness, a compression set, thermal conductivity, and the cell state of the section were measured, and it described in Table 1.

[0040]

[Comparative example 2]

50 copies of side-chain vinyl group content dimethylpolysiloxane (the degree of polymerization 400, vinyl value 0.000094mol/g), 50 copies of dimethylpolysiloxane by which both ends were blocked by the dimethylvinyl siloxy group (degree of polymerization 180), the fumed silica (the product made by Japanese Aerosil.) whose specific surface area is 190m<sup>2</sup>/g Aerosil 200 12 copy, three copies of polyether denaturation silicone shown by the above-mentioned formula (II), 7.0 copies (silicon atom absorbed water matter atom / alkenyl-group (mole ratio) =2.2) of methyl hydrogen polysiloxane of the working example 1 was put into the planetary mixer as a cross linking agent, and stirring was performed for 15 minutes. Then, 60 copies of water was added in 3 steps, and stirring was continued for total 1 hour. Finally, 0.05 copy of ethynylcyclohexanol was added as a reaction controlling agent, and the constituent done for churning continuously [ the constituent / for 15 minutes ] was used as the silicone rubber composition (7).

0.1 copy of platinum catalyst (1% of Pt concentration) was mixed to this silicone rubber composition (7), like the working example 1, it was made to harden, specific gravity, hardness, a compression set, thermal conductivity, and the cell state of the section were measured, and it described in Table 1.

[0041]

[Table 1]

	実施例 1	実施例 2	実施例 3	実施例4	実施例5	比較例 1	比較例2
比重 23℃	0.61	0.72	0.74	0.65	0.63	1.01	0.65
硬度 (デュロメータ-A)	12	17	23	15	16	28	27
圧縮永久歪 (%) [150℃×22時間、25%圧縮]	17	13	29	24	25	7	62
熱伝導率 (W/m・℃)	0.10	0.13	0.13	0.11	0.11	0.19	0.11
セル状態	約5 $\mu$ mの 均一なセル	約5 $\mu$ mの 均一なセル	約5 $\mu$ mの 均一なセル	約10 $\mu$ mのセル であるが、 30 $\mu$ m以上の ものも多数	約10 $\mu$ mの 均一なセル	発泡しな いため セルが存在 しない	約10 $\mu$ mのセル であるが、 50 $\mu$ m以上の ものも多数

[0042]

[Working example 6]

On the surface of the aluminum shaft with a 50 mm[ in diameter ] x length of 30 mm, primer No.101 A/B for addition reaction type liquid silicone rubber (made by Shin-Etsu Chemical Co., Ltd.) was painted. In the metallic mold which installed this aluminum shaft, the silicone rubber composition (1) of the working example 1 was filled up with what added the catalyst, and heat cure was carried out at 95 \*\* for 1 hour, and also postcure was carried out at 180 \*\* for 4 hours, and the silicon rubber roller with an outer diameter [ of 26 mm ] x length of 250 mm was obtained. The surface of this silicon rubber roller was ground, the PFA tube with a thickness of 50 micrometers which applied adhesives to the inner surface was put on the roller surface, it hardened at 180 \*\* for 2 hours, and the PFA resin coating silicon rubber roller was produced.

When this roller was picked out from 180 \*\* oven and it allowed to stand for 2 minutes at the room temperature, roller skin temperature was 72 \*\*. It incorporated as a fixing roller of a PPC copying machine, the heater of after operation and a heater roller was usually stopped, and passage of sheets of 50 sheets was possible.

[0043]

[Comparative example 3]

On the surface of the aluminum shaft with a 50 mm[ in diameter ] x length of 30 mm, primer No.101 A/B for addition reaction type liquid silicone rubber (made by Shin-Etsu Chemical Co., Ltd.) was painted. In the metallic mold which installed this aluminum shaft, the silicone rubber composition (6) of the comparative example 1 was filled up with what added the catalyst, and heat cure was carried out at 95 \*\* for 1 hour, and also postcure was carried out at 180 \*\* for 4 hours, and the silicon rubber roller with an outer diameter [ of 26 mm ] x length of 250 mm was obtained. The surface of this silicon rubber roller was ground, the PFA tube with a thickness of 50 micrometers which applied adhesives to the inner surface was put on the roller surface, it hardened at 180 \*\* for 2 hours, and the PFA resin coating silicon rubber roller was produced.

When this roller was picked out from 180 \*\* oven and it allowed to stand for 2 minutes at the room temperature, roller skin temperature was 46 \*\*. It incorporated as a fixing roller of a PPC copying machine, and after operation, when the heater of the heater roller was stopped and passage of

sheets was continued, a blot usually began to occur because of the insufficiency of fixing from the 25th sheet.

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[Translation done.]

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TECHNICAL FIELD

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[Field of the Invention]

[0001]

This invention relates to a silicone rubber sponge plasticity constituent for fixing rollers of the heat fixing equipment in electrostatic recording devices, such as an electrophotography copying machine, a printer, and a facsimile, a fixing roller formed using this constituent, and a manufacturing method for the same.

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**PRIOR ART**

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**[Background of the Invention]**  
**[0002]**

A heat cure type liquid silicone rubber composition is excellent in a moldability, and after shaping is used in various fields from excelling in heat resistance and electric insulation. In it, since it excels in heat resistance or a mold-release characteristic, it is used for fixing rollers, such as PPC, LBP, and FAX. In the apparatus using these electrophotography processes, it is necessary to fix to tracing paper the toner image transferred by tracing paper from the photo conductor surface. As a method of fixing this toner image, tracing paper is passed between the heater roll and pressure roll which are carrying out contact rotation mutually and which were heated, thermal melting arrival carries out the toner image on tracing paper, and the method of fixing is adopted widely. Although it can be considered as the quick copying machine of a response, a printer, etc. by generally making the thermal conductivity of a roll material high in this thermal melting arrival method, On the other hand, as for the thermally conductive high thing, as for heat dissipation, a good material of heat storage capability is early needed conversely low [ thermal conductivity ] in the flow of a miniaturization and low-pricing. There is silicone rubber foam which used gaseous low-thermal conductivity as this material, and there are a method of using the hydrogen gas which carries out a byproduction as a process of this foam at the time of the method of adding a heat decomposition type blowing agent or hardening, etc.

**[0003]**

However, the method of adding a heat decomposition type blowing agent lets the toxicity of the cracked gas, and a smell be problems.

Hardening inhibition by a foaming agent was made into the problem in what uses a platinum catalyst for a curing catalyst.

In the method of using the hydrogen gas which carries out a byproduction at the time of hardening, there were problems, such as requiring cautions for the handling at the time of the explosivity of hydrogen gas and preservation of uncured material. In shaping made to foam within a metallic mold like injection moulding, there was a problem that it was difficult to obtain the silicone rubber foam which has a minute and uniform cell.

**[0004]**

Although the method of blending a hollow filler was shown by JP,2000-143986,A (patent documents 1), there was a problem that a hollow filler was dramatically lightweight and combination was difficult. By JP,H6-207038,A (patent documents 2), after adding an emulsifier and water to the addition hardening setup-of-tooling product of silicone and making it distribute water, the method of volatilizing water and obtaining sponge is indicated, but about a fixing roller use, it is not suggested at all. Although silica impalpable powder etc. are used as the essential ingredient as a viscous agent, it is not preferred as an object for fixing rollers to blend these so much, in order to worsen a compression set.

[0005]

[Patent documents 1] JP,2000-143986,A

[Patent documents 2] JP,H6-207038,A

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**EFFECT OF THE INVENTION**

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[Effect of the Invention]

[0009]

The fixing roller which formed the cell detailed [ the silicone rubber sponge plasticity constituent for fixing rollers of this invention ] and uniform, thermal conductivity was low, could become a hardened material which is excellent in endurance, and was formed using this is low-feeve conduction, and is excellent in endurance.

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**TECHNICAL PROBLEM**

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[Problem to be solved by the invention]  
[0006]

This invention was made in view of the above-mentioned situation, and forms a detailed and uniform cell, and an object of this invention is to provide a silicone rubber sponge plasticity constituent for fixing rollers which gives the hardened material which is low-feeve conduction and was excellent in endurance, a fixing roller formed using this constituent, and a manufacturing method for the same.

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MEANS

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[Means for solving problem]

[0007]

this invention person found out that the obtained silicone rubber sponge was suitable as a fixing roller use by blending a surface-active agent and water with an addition hardening type silicone rubber composition, as a result of inquiring wholeheartedly, in order to attain the above-mentioned purpose.

Namely, the liquefied organopolysiloxane which contains the alkenyl group combined with at least two silicon atoms in the (A) monad, (B) The liquefied ORGANO hydrogen polysiloxane which contains in a monad the hydrogen atom combined with at least two silicon atoms, (C) By using a surface-active agent and the silicone rubber sponge plasticity constituent for fixing rollers which contains the specific amount of polyether denaturation silicone, the (D) addition reaction catalyst, (E) reinforcement nature silica impalpable powder, and (F) water preferably, A detailed and uniform cell is formed, and it finds out that the fixing roller which is low-fever conduction and was excellent in endurance can be manufactured, and came to make this invention.

[0008]

Therefore, this invention provides a silicone rubber sponge plasticity constituent for the following fixing rollers, a fixing roller, and a manufacturing method for the same.

[1] Liquefied organopolysiloxane 100 mass part which contains the alkenyl group combined with at least two silicon atoms in the (A) monad

(B) 0.1 to liquefied ORGANO hydrogen polysiloxane 30 mass part which contains in a monad the hydrogen atom combined with at least two silicon atoms

(C) One to surface-active agent 20 mass part

(D) Addition reaction catalyst Catalyst amount

(E) Zero to reinforcement nature silica impalpable powder 10 mass part

(F) 30 to water 200 mass part

The silicone rubber sponge plasticity constituent for fixing rollers to contain.

[2] It is a heat fusing roller with which it comes to form a silicone rubber sponge layer in the peripheral face of roll axes, The silicone rubber sponge fixing roller with which the silicone rubber sponge which forms this silicone rubber sponge layer is characterized by making it come to harden the above-mentioned silicone rubber sponge plasticity constituent.

[3] A manufacturing method of a silicone rubber sponge fixing roller which makes a silicone rubber sponge layer form in it by volatilizing moisture at temperature of not less than 100 \*\* after making a peripheral face of roll axes harden the above-mentioned silicone rubber sponge plasticity constituent at less than 100 \*\*.

[4] It is a fluororesin coating fixing roller with which it comes to form a fluoro-resin or fluororubber layers in a peripheral face of roll axes via a silicone rubber sponge layer, A fluoro-resin or a fluorocarbon rubber covering silicone rubber sponge fixing roller with which silicone rubber sponge

which forms this silicone rubber sponge layer is characterized by making it come to harden the above-mentioned silicone rubber sponge plasticity constituent.

[5] A peripheral face of roll axes is made to harden the above-mentioned silicone rubber sponge plasticity constituent at less than 100 \*\*, A manufacturing method of a fluoro-resin which grinds the silicone rubber sponge layer surface and makes a fluoro-resin or fluororubber layers form in this surface after making a silicone rubber sponge layer form by volatilizing moisture at temperature of not less than 100 \*\*, or a fluorocarbon rubber covering silicone rubber sponge fixing roller.

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## EXAMPLE

[Working example]

[0033]

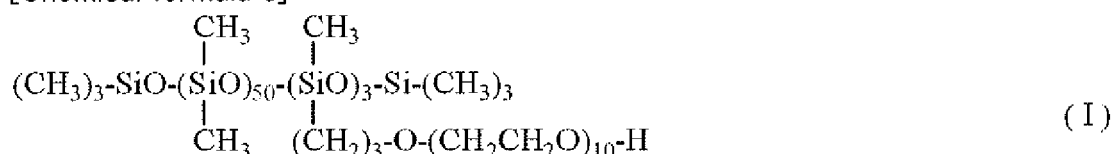
Although an working example and a comparative example are shown and this invention is explained concretely hereafter, this invention is not restricted to the following working example. In the following example, a part and % show mass part and mass %, respectively. A molecular weight is the weight average molecular weight of the polystyrene conversion by GPC.

[0034]

[Working example 1]

100 copies of dimethylpolysiloxane (the degree of polymerization 220, vinyl value 0.000125 mol/g) by which both ends were blocked by the dimethylvinyl siloxy group, three copies of fumed silica (the product made by Japanese Aerosil, R-972) whose specific surface area is 110m<sup>2</sup>/g and by which hydrophobing processing was carried out, following formula (I)

[Chemical formula 3]



Come out and eight copies of polyether denaturation silicone (the molecular weight 5540, 73% of the amount of siloxanes in a molecule) shown is put into a planetary mixer, After continuing churning for 10 minutes, 4.3 copies (silicon atom absorbed water matter atom / alkenyl-group (mole ratio) =1.3) of methyl hydrogen polysiloxane (the degree of polymerization 18, amount of Si-H 0.0038 mol/g) which has a Si-H basis in both ends and a side chain as a cross linking agent was added, and stirring was performed for 10 minutes. 80 copies of water was added in 4 steps to this, and stirring was continued for total 1 hour. Finally 0.05 copy of ethynylcyclohexanol was added as a reaction controlling agent, and the constituent done for churning continuously [ the constituent / for 15 minutes ] was used as the silicone rubber composition (1).

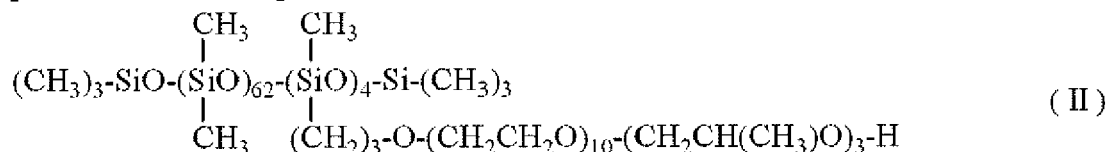
Mix 0.1 copy of platinum catalyst (1% of Pt concentration) to this silicone rubber composition (1), slush into a mold, and it is neglected in 95 \*\* oven for 1 hour, After making it harden, it removed from the mold and described in Table 1 about the result of having measured specific gravity, hardness, and a compression set based on JIS K 6249 about the hardened material obtained by carrying out postcure for 4 hours at 200 \*\*. Similarly it described in Table 1 about the result of having observed the cell state of the section with the electron microscope as a result of measuring thermal conductivity with a heat-conduction plan (QTM-3 Kyoto electronic company make).

[0035]

[An working example 2]

50 copies of side-chain vinyl group content dimethylpolysiloxane (the degree of polymerization 400, vinyl value 0.000094mol/g), 50 copies of dimethylpolysiloxane (degree of polymerization 180) by which both ends were blocked by a dimethylvinyl siloxy group, 0.5 copy of fumed silica (a product made by Japanese Aerosil, Aerosil 200) whose specific surface area is 190m<sup>2</sup>/g, following formula (II)

[Chemical formula 4]



Three copies of polyether denaturation silicone come out of and shown (the molecular weight 5460, 51% of the amount of siloxanes in a molecule), 3.2 copies (silicon atom absorbed water matter atom / alkenyl-group (mole ratio) =1.0) were put into a planetary mixer for methyl hydrogen polysiloxane of an working example 1 as a cross linking agent, and stirring was performed for 15 minutes. Then, 60 copies of water was added in 3 steps, and stirring was continued for total 1 hour. Finally, 0.05 copy of ethynylcyclohexanol was added as a reaction controlling agent, and a constituent done for churning continuously [ a constituent / for 15 minutes ] was used as a silicone rubber composition (2).

0.1 copy of platinum catalyst (1% of Pt concentration) was mixed to this silicone rubber composition (2), like an working example 1, it was made to harden, specific gravity, hardness, a compression set, thermal conductivity, and a cell state of a section were measured, and it described in Table 1.

[0036]

[Working example 3]

50 copies of side-chain vinyl group content dimethylpolysiloxane (the degree of polymerization 400, vinyl value 0.000094mol/g), 50 copies of dimethylpolysiloxane by which both ends were blocked by the dimethylvinyl siloxy group (degree of polymerization 180), the fumed silica (the product made by Japanese Aerosil.) whose specific surface area is 190m<sup>2</sup>/g Aerosil 200 0.5 copy, three copies of polyether denaturation silicone shown by the above-mentioned formula (II), 7.0 copies (silicon atom absorbed water matter atom / alkenyl-group (mole ratio) =2.2) of methyl hydrogen polysiloxane of the working example 1 was put into the planetary mixer as a cross linking agent, and stirring was performed for 15 minutes. Then, 60 copies of water was added in 3 steps, and stirring was continued for total 1 hour. Finally, 0.05 copy of ethynylcyclohexanol was added as a reaction controlling agent, and the constituent done for churning continuously [ the constituent / for 15 minutes ] was used as the silicone rubber composition (3).

0.1 copy of platinum catalyst (1% of Pt concentration) was mixed to this silicone rubber composition (3), like the working example 1, it was made to harden, specific gravity, hardness, a compression set, thermal conductivity, and the cell state of the section were measured, and it described in Table 1.

[0037]

[Working example 4]

100 copies of dimethylpolysiloxane which both ends are blocked by a dimethylvinyl siloxy group, and contains a vinyl group in a side chain (the degree of polymerization 550, vinyl value 0.000120 mol/g), the fumed silica (the product made by Japanese Aerosil.) whose specific surface area is 110m<sup>2</sup>/g and by which hydrophobing processing was carried out R-972) Put eight copies of polyether denaturation silicone shown by three copies and the above-mentioned formula (I) into a planetary mixer. After continuing churning for 10 minutes, 4.1 copies (silicon atom absorbed water matter atom / alkenyl-group (mole ratio) =1.3) of methyl hydrogen polysiloxane of the working example 1 was added, and stirring was performed for 10 minutes. 80 copies of water was added in 4 steps to



this, and stirring was continued for total 1 hour. Finally 0.05 copy of ethynylcyclohexanol was added as a reaction controlling agent, and the constituent done for churning continuously [ the constituent / for 15 minutes ] was used as the silicone rubber composition (4).

0.1 copy of platinum catalyst (1% of Pt concentration) was mixed to this silicone rubber composition (4), like the working example 1, it was made to harden, specific gravity, hardness, a compression set, thermal conductivity, and the cell state of the section were measured, and it described in Table 1.

[0038]

[Working example 5]

100 copies of dimethylpolysiloxane of the working example 1 by which both ends were blocked by the dimethylvinyl siloxy group (the degree of polymerization 220, vinyl value 0.0125mol/100 g), Three copies of fumed silica (the product made by Japanese Aerosil, R-972), following formula (III) whose specific surface area is 110m<sup>2</sup>/g and by which hydrophobing processing was carried out

[Chemical formula 5]



After having come out, putting eight copies of non-ion system surface-active agents shown into the planetary mixer and continuing churning for 10 minutes, 4.3 copies of methyl hydrogen polysiloxane of the working example 1 was added as a cross linking agent, and stirring was performed for 10 minutes. 80 copies of water was added in 4 steps to this, and stirring was continued for total 1 hour. Finally 0.05 copy of ethynylcyclohexanol was added as a reaction controlling agent, and the constituent done for churning continuously [ the constituent / for 15 minutes ] was used as the silicone rubber composition (5).

0.1 copy of platinum catalyst (1% of Pt concentration) was mixed to this silicone rubber composition (5), like the working example 1, it was made to harden, specific gravity, hardness, a compression set, thermal conductivity, and the cell state of the section were measured, and it described in Table 1.

[0039]

[Comparative example 1]

100 copies of dimethylpolysiloxane by which the both ends of the working example 1 were blocked by the dimethylvinyl siloxy group (the degree of polymerization 220, vinyl value 0.0125mol/100 g), the fumed silica (the product made by Japanese Aerosil.) whose specific surface area is 110m<sup>2</sup>/g and by which hydrophobing processing was carried out R-972) After putting eight copies of polyether denaturation silicone shown by three copies and the above-mentioned formula (I) into the planetary mixer and continuing churning for 10 minutes, 4.3 copies of methyl hydrogen polysiloxane of the working example 1 was added as a cross linking agent, and stirring was performed for 10 minutes. Finally 0.05 copy of ethynylcyclohexanol was added as a reaction controlling agent, and the constituent done for churning continuously [ the constituent / for 15 minutes ] was used as the silicone rubber composition (6).

0.1 copy of platinum catalyst (1% of Pt concentration) was mixed to this silicone rubber composition (6), like the working example 1, it was made to harden, specific gravity, hardness, a compression set, thermal conductivity, and the cell state of the section were measured, and it described in Table 1.

[0040]

[Comparative example 2]

50 copies of side-chain vinyl group content dimethylpolysiloxane (the degree of polymerization 400, vinyl value 0.000094 mol/g), 50 copies of dimethylpolysiloxane by which both ends were blocked by the dimethylvinyl siloxy group (degree of polymerization 180), the fumed silica (the product made by Japanese Aerosil.) whose specific surface area is 190m<sup>2</sup>/g Aerosil 200 12 copy, three copies of polyether denaturation silicone shown by the above-mentioned formula (II), 7.0 copies (silicon atom absorbed water matter atom / alkenyl-group (mole ratio) =2.2) of methyl hydrogen polysiloxane of

the working example 1 was put into the planetary mixer as a cross linking agent, and stirring was performed for 15 minutes. Then, 60 copies of water was added in 3 steps, and stirring was continued for total 1 hour. Finally, 0.05 copy of ethynylcyclohexanol was added as a reaction controlling agent, and the constituent done for churning continuously [ the constituent / for 15 minutes ] was used as the silicone rubber composition (7).

0.1 copy of platinum catalyst (1% of Pt concentration) was mixed to this silicone rubber composition (7), like the working example 1, it was made to harden, specific gravity, hardness, a compression set, thermal conductivity, and the cell state of the section were measured, and it described in Table 1.

[0041]

[Table 1]

	実施例 1	実施例 2	実施例 3	実施例4	実施例5	比較例 1	比較例2
比重 23℃	0.61	0.72	0.74	0.65	0.63	1.01	0.65
硬度 (デュロメータ-A)	12	17	23	15	16	28	27
圧縮永久歪(%) [150℃×22時間、25%圧縮]	17	13	29	24	25	7	62
熱伝導率 (W/m・℃)	0.10	0.13	0.13	0.11	0.11	0.19	0.11
セル状態	約5 $\mu$ mの 均一なセル	約5 $\mu$ mの 均一なセル	約5 $\mu$ mの 均一なセル	約10 $\mu$ mのセル であるが、 30 $\mu$ m以上の ものも多数	約10 $\mu$ mの 均一なセル	発泡しな いため セルが存在 しない	約10 $\mu$ mのセル であるが、 50 $\mu$ m以上の ものも多数

[0042]

[Working example 6]

On the surface of the aluminum shaft with a 50 mm[ in diameter ] x length of 30 mm, primer No.101 A/B for addition reaction type liquid silicone rubber (made by Shin-Etsu Chemical Co., Ltd.) was painted. In the metallic mold which installed this aluminum shaft, the silicone rubber composition (1) of the working example 1 was filled up with what added the catalyst, and heat cure was carried out at 95 \*\* for 1 hour, and also postcure was carried out at 180 \*\* for 4 hours, and the silicon rubber roller with an outer diameter [ of 26 mm ] x length of 250 mm was obtained. The surface of this silicon rubber roller was ground, the PFA tube with a thickness of 50 micrometers which applied adhesives to the inner surface was put on the roller surface, it hardened at 180 \*\* for 2 hours, and the PFA resin coating silicon rubber roller was produced.

When this roller was picked out from 180 \*\* oven and it allowed to stand for 2 minutes at the room temperature, roller skin temperature was 72 \*\*. It incorporated as a fixing roller of a PPC copying machine, the heater of after operation and a heater roller was usually stopped, and passage of sheets of 50 sheets was possible.

[0043]

[Comparative example 3]

On the surface of the aluminum shaft with a 50 mm[ in diameter ] x length of 30 mm, primer No.101 A/B for addition reaction type liquid silicone rubber (made by Shin-Etsu Chemical Co., Ltd.) was

painted. In the metallic mold which installed this aluminum shaft, the silicone rubber composition (6) of the comparative example 1 was filled up with what added the catalyst, and heat cure was carried out at 95 \*\* for 1 hour, and also postcure was carried out at 180 \*\* for 4 hours, and the silicon rubber roller with an outer diameter [ of 26 mm ] x length of 250 mm was obtained. The surface of this silicon rubber roller was ground, the PFA tube with a thickness of 50 micrometers which applied adhesives to the inner surface was put on the roller surface, it hardened at 180 \*\* for 2 hours, and the PFA resin coating silicon rubber roller was produced.

When this roller was picked out from 180 \*\* oven and it allowed to stand for 2 minutes at the room temperature, roller skin temperature was 46 \*\*. It incorporated as a fixing roller of a PPC copying machine, and after operation, when the heater of the heater roller was stopped and passage of sheets was continued, a blot usually began to occur because of the insufficiency of fixing from the 25th sheet.

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[Translation done.]

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(54) 【発明の名称】 定着ローラ用シリコンゴムスポンジ形成性組成物、定着ローラ及びその製造方法

## (57) 【要約】

【課題】 (A) 一分子中に少なくとも2個の珪素原子と結合するアルケニル基を含有する液状オルガノポリシロキサン 100質量部

(B) 一分子中に少なくとも2個の珪素原子と結合する水素原子を含有する液状オルガノハイドロジェンポリシロキサン 0、1～30質量部

(C) 界面活性剤 1～20質量部

(D) 付加反応触媒 触媒量

(E) 補強性シリカ微粉末 0～10質量部

(F) 水 30～200質量部

を含有する定着ローラ用シリコンゴムスポンジ形成性組成物、この組成物を用いて形成された定着ローラ及びその製造方法。 10

【解決手段】 本発明の定着ローラ用シリコンゴムスポンジ形成性組成物は、微細で均一なセルを形成し、熱伝導性が低く、耐久性に優れた硬化物となり得、これを用いて形成された定着ローラは、低熱伝導かつ耐久性に優れたものである。

【選択図】 なし

(2)

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## 【特許請求の範囲】

## 【請求項 1】

- (A) 一分子中に少なくとも 2 個の珪素原子と結合するアルケニル基を含有する液状オルガノポリシロキサン 100 質量部  
 (B) 一分子中に少なくとも 2 個の珪素原子と結合する水素原子を含有する液状オルガノ  
 ハイドロジェンポリシロキサン 0.1～30 質量部  
 (C) 界面活性剤 1～20 質量部  
 (D) 付加反応触媒 触媒量  
 (E) 補強性シリカ微粉末 0～10 質量部  
 (F) 水 30～200 質量部 10

を含有する定着ローラ用シリコーンゴムスポンジ形成性組成物。

## 【請求項 2】

(A) 成分のアルケニル基と (B) 成分の珪素原子結合水素原子とのモル比 (Si-H 基/アルケニル基) が、2.0 以下である請求項 1 記載の定着ローラ用シリコーンゴムスポンジ形成性組成物。

## 【請求項 3】

(A) 成分のオルガノポリシロキサンの重合度が、400 以下である請求項 1 又は 2 記載の定着ローラ用シリコーンゴムスポンジ形成性組成物。

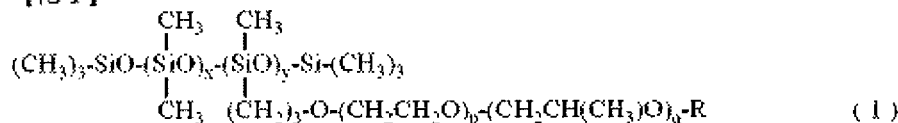
## 【請求項 4】

(C) 成分の界面活性剤が、ポリエーテル変性シリコーンである請求項 1、2 又は 3 記載の定着ローラ用シリコーンゴムスポンジ形成性組成物。 20

## 【請求項 5】

ポリエーテル変性シリコーンが、下記式 (1) で表されるものである請求項 4 記載の定着ローラ用シリコーンゴムスポンジ形成性組成物。

## 【化 1】



[式中、R は H 又は C H<sub>3</sub> であり、x は 10～200、y は 1～20 で、かつ x/y は 5～50 を満足する整数であり、p は 3～50、q は 0～30 で、かつ p/(p+q) は 0.5～1 を満足する整数である。また、分子量は 2,000～50,000 であり、分子中のシロキサン量は 40～95 質量%である。]

## 【請求項 6】

硬化物の熱伝導率が、0.15 W/m・℃以下である請求項 1 乃至 5 のいずれか 1 項記載の定着ローラ用シリコーンゴムスポンジ形成性組成物。

## 【請求項 7】

ロール軸の外周面にシリコーンゴムスポンジ層が形成されてなる熱定着ローラであって、このシリコーンゴムスポンジ層を形成するシリコーンゴムスポンジが、請求項 1 乃至 6 のいずれか 1 項記載のシリコーンゴムスポンジ形成性組成物を硬化させてなることを特徴とするシリコーンゴムスポンジ定着ローラ。 40

## 【請求項 8】

ロール軸の外周面に、請求項 1 乃至 6 のいずれか 1 項記載のシリコーンゴムスポンジ形成性組成物を 100℃未満で硬化させた後、100℃以上の温度で水分を揮発させることによりシリコーンゴムスポンジ層を形成させるシリコーンゴムスポンジ定着ローラの製造方法。

## 【請求項 9】

ロール軸の外周面にシリコーンゴムスポンジ層を介してフッ素樹脂又はフッ素ゴム層が形成されてなるフッ素樹脂被覆定着ローラであって、このシリコーンゴムスポンジ層を形 50

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成するシリコンゴムスポンジが、請求項 1 乃至 6 のいずれか 1 項記載のシリコンゴムスポンジ形成性組成物を硬化させてなることを特徴とするフッ素樹脂又はフッ素ゴム被覆シリコンゴムスポンジ定着ローラ。

#### 【請求項 10】

ロール軸の外周面に、請求項 1 乃至 6 のいずれか 1 項記載のシリコンゴムスポンジ形成性組成物を 100℃未満で硬化させ、100℃以上の温度で水分を揮発させることによりシリコンゴムスポンジ層を形成させた後、更にシリコンゴムスポンジ層表面を研磨し、該表面にフッ素樹脂又はフッ素ゴム層を形成させるフッ素樹脂又はフッ素ゴム被覆シリコンゴムスポンジ定着ローラの製造方法。

#### 【発明の詳細な説明】

#### 【技術分野】

#### 【0001】

本発明は、電子写真複写機、プリンター、ファクシミリ等の静電記録装置における加熱定着装置の定着ローラ用シリコンゴムスポンジ形成性組成物、この組成物を用いて形成された定着ローラ及びその製造方法に関するものである。

#### 【背景技術】

#### 【0002】

加熱硬化型液状シリコンゴム組成物は成形性に優れ、成形後は耐熱性、電気絶縁性に優れることから種々の分野で使用されている。その中で、耐熱性や離型性に優れることから P P C、L B P、F A X 等の定着ローラに使用されている。これら電子写真プロセスを利用した機器においては、感光体表面から複写紙に転写されたトナー像を複写紙に固定する必要がある。このトナー像を固定する方法として、互いに圧接回転している加熱されたヒーターロールと加圧ロールとの間に複写紙を通過させ、複写紙上のトナー像を熱融着し、固定する方法が広く採用されている。この熱融着方法においては、一般にロール材料の熱伝導率を高くすることで、応答の速い複写機、プリンターなどとすることができるが、一方で熱伝導性の高いものは放熱も早く、小型化、低価格化の流れの中で、逆に熱伝導性の低い、すなわち蓄熱性のよい材料が必要とされている。かかる材料として気体の低熱伝導性を利用したシリコンゴム発泡体があり、この発泡体の製法としては、熱分解型発泡剤を添加する方法や硬化時に副生する水素ガスを利用する方法などがある。

#### 【0003】

ところが、熱分解型発泡剤を添加する方法は、その分解ガスの毒性や臭いが問題点とされており、また硬化触媒に白金触媒を使用するものでは発泡剤による硬化阻害が問題とされていた。また、硬化時に副生する水素ガスを利用する方法においては、水素ガスの爆発性、未硬化物の保存時の取り扱いに注意を要するなどの問題があった。更に射出成形のように金型内で発泡させる成形においては、微小かつ均一なセルを有するシリコンゴム発泡体を得ることが難しいという問題があった。

#### 【0004】

また、特開 2000-143986 号公報（特許文献 1）では、中空フィラーを配合する方法が示されているが、中空フィラーが非常に軽量で配合が困難であるという問題があった。特開平 6-207038 号公報（特許文献 2）では、シリコンの付加硬化型組成物に乳化剤と水を添加して水を分散させた後、水を揮発させてスポンジを得る方法が記載されているが、定着ローラ用途については全く示唆されていない。更に、粘糊剤としてシリカ微粉末などが必須成分とされているが、これらを多量に配合することは、圧縮永久歪を悪化させるため、定着ローラ用としては好ましくない。

#### 【0005】

【特許文献 1】特開 2000-143986 号公報

【特許文献 2】特開平 6-207038 号公報

#### 【発明の開示】

#### 【発明が解決しようとする課題】

#### 【0006】

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本発明は、上記事情に鑑みなされたもので、微細で均一なセルを形成し、低熱伝導でかつ耐久性に優れた硬化物を与える定着ローラ用シリコーンゴムスポンジ形成性組成物、この組成物を用いて形成された定着ローラ及びその製造方法を提供することを目的とする。

【課題を解決するための手段】

【0007】

本発明者は、上記目的を達成するため鋭意検討した結果、付加硬化型シリコーンゴム組成物に界面活性剤及び水を配合することにより、得られたシリコーンゴムスポンジが、定着ローラ用途として適することを見出した。

即ち、(A) 一分子中に少なくとも2個の珪素原子と結合するアルケニル基を含有する液状オルガノポリシロキサン、(B) 一分子中に少なくとも2個の珪素原子と結合する水素原子を含有する液状オルガノハイドロジェンポリシロキサン、(C) 界面活性剤、好ましくはポリエーテル変性シリコーン、(D) 付加反応触媒、(E) 補強性シリカ微粉末、及び(F) 水の特定量を含有する定着ローラ用シリコーンゴムスポンジ形成性組成物を用いることにより、微細で均一なセルを形成し、低熱伝導でかつ耐久性に優れた定着ローラを製造し得ることを見出し、本発明をなすに至った。

【0008】

従って、本発明は、下記定着ローラ用シリコーンゴムスポンジ形成性組成物、定着ローラ及びその製造方法を提供する。

(1) (A) 一分子中に少なくとも2個の珪素原子と結合するアルケニル基を含有する液状オルガノポリシロキサン	100質量部	20
(B) 一分子中に少なくとも2個の珪素原子と結合する水素原子を含有する液状オルガノハイドロジェンポリシロキサン	0.1～30質量部	
(C) 界面活性剤	1～20質量部	
(D) 付加反応触媒	触媒量	
(E) 補強性シリカ微粉末	0～10質量部	
(F) 水	30～200質量部	

を含有する定着ローラ用シリコーンゴムスポンジ形成性組成物。

【2】 ロール軸の外周面にシリコーンゴムスポンジ層が形成されてなる熱定着ローラであって、このシリコーンゴムスポンジ層を形成するシリコーンゴムスポンジが、上記シリコーンゴムスポンジ形成性組成物を硬化させてなることを特徴とするシリコーンゴムスポンジ定着ローラ。

【3】 ロール軸の外周面に、上記シリコーンゴムスポンジ形成性組成物を100℃未満で硬化させた後、100℃以上の温度で水分を揮発させることによりシリコーンゴムスポンジ層を形成させるシリコーンゴムスポンジ定着ローラの製造方法。

【4】 ロール軸の外周面にシリコーンゴムスポンジ層を介してフッ素樹脂又はフッ素ゴム層が形成されてなるフッ素樹脂被覆定着ローラであって、このシリコーンゴムスポンジ層を形成するシリコーンゴムスポンジが、上記シリコーンゴムスポンジ形成性組成物を硬化させてなることを特徴とするフッ素樹脂又はフッ素ゴム被覆シリコーンゴムスポンジ定着ローラ。

【5】 ロール軸の外周面に、上記シリコーンゴムスポンジ形成性組成物を100℃未満で硬化させ、100℃以上の温度で水分を揮発させることによりシリコーンゴムスポンジ層を形成させた後、更にシリコーンゴムスポンジ層表面を研磨し、該表面にフッ素樹脂又はフッ素ゴム層を形成させるフッ素樹脂又はフッ素ゴム被覆シリコーンゴムスポンジ定着ローラの製造方法。

【発明の効果】

【0009】

本発明の定着ローラ用シリコーンゴムスポンジ形成性組成物は、微細で均一なセルを形成し、熱伝導性が低く、耐久性に優れた硬化物となり得、これを用いて形成された定着ローラは、低熱伝導でかつ耐久性に優れたものである。

【発明を実施するための最良の形態】

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## 【0010】

本発明を更に詳しく説明すると、本発明の定着ローラ用シリコーンゴムスポンジ形成性組成物は、下記(A)～(F)成分を含有するものである。

(A) 一分子中に少なくとも2個の珪素原子と結合するアルケニル基を含有する液状オルガノポリシロキサン	100質量部	
(B) 一分子中に少なくとも2個の珪素原子と結合する水素原子を含有する液状オルガノハイドロジェンポリシロキサン	0、1～30質量部	
(C) 界面活性剤	1～20質量部	
(D) 付加反応触媒	触媒量	
(E) 補強性シリカ微粉末	0～10質量部	10
(F) 水	30～200質量部	

## 【0011】

(A)成分の一分子中に少なくとも2個の珪素原子と結合するアルケニル基を含有する液状オルガノポリシロキサンは、本組成物の主剤（ベースポリマー）であり、このアルケニル基含有オルガノポリシロキサンとしては、下記平均組成式(2)で示されるものを用いることができる。



## 【0012】

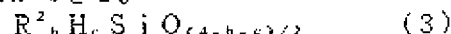
式中、 $R^1$ は互いに同一又は異種の炭素数1～10、好ましくは1～8の非置換又は置換一価炭化水素基であり、 $a$ は1、5～2、8、好ましくは1、8～2、5、より好ましくは1、95～2、02の範囲の正数である。ここで、上記 $R^1$ で示されるケイ素原子に結合した非置換又は置換の一価炭化水素基としては、メチル基、エチル基、プロピル基、イソプロピル基、ブチル基、イソブチル基、tert-ブチル基、ペンチル基、ネオペンチル基、ヘキシル基、シクロヘキシル基、オクチル基、ノニル基、デシル基等のアルキル基、フェニル基、トリル基、キシリル基、ナフチル基等のアリール基、ベンジル基、フェニルエチル基、フェニルプロピル基等のアラールキル基、ビニル基、アリル基、プロペニル基、イソプロペニル基、ブテニル基、ヘキセニル基、シクロヘキセニル基、オクテニル基等のアルケニル基などや、これらの基の水素原子の一部又は全部をフッ素、臭素、塩素等のハロゲン原子、シアノ基等で置換したもの、例えばクロロメチル基、クロロプロピル基、ブロモエチル基、トリフロロプロピル基、シアノエチル基等が挙げられる。この場合、 $R^1$ のうち少なくとも2個はアルケニル基（炭素数2～8のものが好ましく、更に好ましくは2～6のものである。）であることが必要である。なお、アルケニル基の含有量は、 $R^1$ 中0、001～20モル％、特に0、01～10モル％であることが好ましい。このアルケニル基は、分子鎖末端のケイ素原子に結合していても、分子鎖途中のケイ素原子に結合していても、両者に結合していてもよい。

## 【0013】

このオルガノポリシロキサンの構造は、通常、主鎖がジオルガノシロキサン単位の繰り返しからなり、分子鎖両末端がトリオルガノシロキシ基で封鎖された基本的には直鎖状構造を有するが、部分的には分岐状の構造、環状構造などであってもよい。重合度（又は1分子中の珪素原子の数）については、室温で液状であればよいが、高粘度のものは水が分散しにくくなるため、通常、重合度が800以下、好ましくは重合度が400以下、より好ましくは重合度が300以下である。その下限は限定されるものではないが、通常、重合度が50以上、好ましくは重合度が100以上のものである。

## 【0014】

(B)成分の液状オルガノハイドロジェンポリシロキサンは、一分子中に少なくとも2個、好ましくは3個以上（通常3～200個）、より好ましくは3～100個のケイ素原子結合水素原子を有することが必要であり、下記平均組成式(3)で示されるものを用いることができる。



(式中、 $R^2$ は炭素数1～10の置換又は非置換の一価炭化水素基である。また $b$ は、0



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、7～2.1、cは0.001～1.0で、かつb+cは0.8～3.0を満足する正数である。)

## 【0015】

ここで、 $R^2$ の一価炭化水素基としては、 $R^1$ で例示したものと同様のものを挙げることができるが、脂肪族不飽和基を有しないものが好ましい。また、bは好ましくは0.8～2.0、cは好ましくは0.01～1.0、b+cは好ましくは1.0～2.5であり、オルガノハイドロジェンポリシロキサン分子構造は、直鎖状、環状、分岐状、三次元網目状のいずれの構造であってもよい。この場合、一分子中の珪素原子の数(又は重合度)は2～300個、特に4～150個程度の室温(25℃)で液状のものが好適に用いられる。なお、珪素原子に結合する水素原子は分子鎖末端、分子鎖の途中のいずれに位置していてもよく、両方に位置するものであってもよい。

## 【0016】

このようなオルガノハイドロジェンポリシロキサンとしては、両末端トリメチルシロキシ基封鎖メチルハイドロジェンポリシロキサン、両末端トリメチルシロキシ基封鎖ジメチルシロキサン・メチルハイドロジェンシロキサン共重合体、両末端ジメチルハイドロジェンシロキシ基封鎖ジメチルポリシロキサン、両末端ジメチルハイドロジェンシロキシ基封鎖ジメチルシロキサン・メチルハイドロジェンシロキサン共重合体、両末端トリメチルシロキシ基封鎖メチルハイドロジェンシロキサン・ジフェニルシロキサン共重合体、両末端トリメチルシロキシ基封鎖メチルハイドロジェンシロキサン・ジフェニルシロキサン・ジメチルシロキサン共重合体、 $(CH_3)_2HSiO_{1/2}$ 単位と $SiO_{2/2}$ 単位とから成る共重合体、 $(CH_3)_2HSiO_{1/2}$ 単位と $SiO_{2/2}$ 単位と $(C_6H_5)_SiO_{3/2}$ 単位とから成る共重合体などが挙げられる。

## 【0017】

このオルガノハイドロジェンポリシロキサンの配合量は、(A)成分のオルガノポリシロキサン100質量部に対して0.1～30質量部であり、好ましくは0.3～20質量部である。配合量が、0.1質量部未満では、硬化が不十分でゲル状になってしまい、ゴム状の弾性体硬化物を与えることができず、30質量部を超える量では、硬化物の強度と耐圧縮永久歪性が著しく低下して(圧縮永久歪が大きくなって)しまう。

また、(A)成分のアルケニル基と珪素原子結合水素原子とのモル比( $Si-H$ 基/アルケニル基)が、好ましくは2.0以下、特に好ましくは0.5～1.6である。この比が2.0を超えると、圧縮永久歪が大きくなるばかりか、脱水素反応が優先し、均一微細なセルが得られなくなってしまうおそれがある。また、この比が小さすぎると、ゴム強度が不十分になってしまうおそれがある。

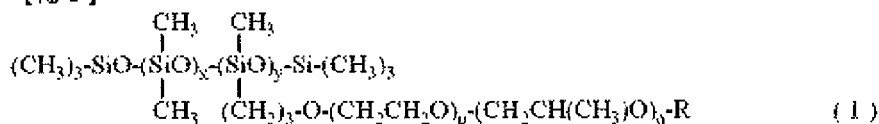
## 【0018】

(C)成分の界面活性剤は、アニオン系、カチオン系、両性イオン系及びノニオン系のいずれでもかまわない。アニオン系界面活性剤としては、例えば、高級脂肪酸塩類、高級アルコール硫酸エステル塩類、アルキルベンゼンスルホン酸塩類、アルキルナフタレンスルホン酸塩類、ポリエチレングリコール硫酸エステル塩類を挙げることができる。またノニオン系界面活性剤としては、例えば、ポリオキシエチレンアルキルフェニルエーテル類、ソルビタン脂肪酸エステル類、ポリオキシエチレンソルビタン脂肪酸エステル類、ポリオキシアルキレン脂肪酸エステル類、ポリオキシエチレンポリオキシプロピレン類、脂肪酸モノグリセライド類を挙げることができる。更にカチオン系界面活性剤としては、例えば、脂肪族アミン塩類、第4級アンモニウム塩類、アルキルピリジニウム塩類を挙げることができる。両性イオン系界面活性剤としては、カルボキシベタイン型、グリシン型のものが挙げられる。これらの中で、ポリエーテル変性シリコンが微細セルで高耐久なスポンジを形成できる点で好ましい。このようなポリエーテル変性シリコンとしては、下記式(1)で表されるものが好適である。

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【化1】



【0019】

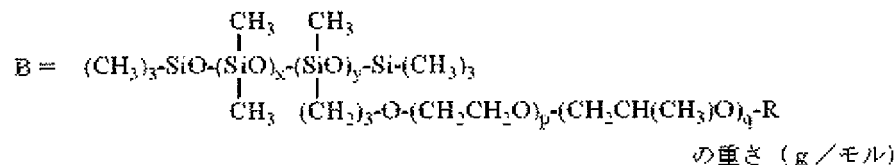
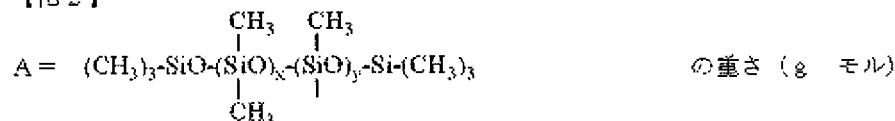
式中、RはH又はCH<sub>3</sub>であり、xは10～200、好ましくは20～100であり、yは1～20、好ましくは2～10であり、x/yは5～50、好ましくは5～30を満足する整数である。pは3～50、好ましくは3～40であり、qは0～30、好ましくは0～20であり、かつp/(p+q)は0.5～1、好ましくは0.7～1を満足する整数である。

【0020】

また、分子量は2,000～50,000、好ましくは2,000～20,000である。この場合、この分子量測定はGPC（ゲルパーミエーションクロマトグラフィー）によるポリスチレン換算の重量平均分子量による値である。更に、分子中のシロキサン量は40～95質量%、好ましくは50～85質量%である。この場合、このシロキサン量は、(A/B)×100（質量%）で示される値である。

【0021】

【化2】



（但し、R、x、y、p、qは上記と同様。）

【0022】

これらの界面活性剤は、単独で使用してもよく、また2種以上を用いてもよい。これら界面活性剤の配合量は、(A)成分のオルガノポリシロキサン100質量部に対し、1～20質量部、好ましくは2～15質量部である。1質量部未満では水の分散が不十分で、微細なセルが得られず、20質量部を超える量ではゴム物性に悪影響を与えてしまう。

【0023】

(D)成分の付加反応触媒としては、白金黒、塩化第2白金、塩化白金酸、塩化白金酸と1価アルコールとの反応物、塩化白金酸とオレフィン類との錯体、白金ビスアセトアセテート等の白金系触媒、パラジウム系触媒、ロジウム系触媒などが挙げられる。なおこの付加反応触媒の配合量は触媒量とすることができ、通常白金族金属として(A)成分に対し、0.5～1,000ppm、特に1～500ppm程度である。

【0024】

(E)成分の補強性シリカ微粉末は、硬化物のゴム強度を得るために添加されるもので、特に限定されないが、ヒュームドシリカ及び沈降シリカが好適である。また、これらのシリカ微粉末は、例えば鎖状オルガノポリシロキサン、環状オルガノポリシロキサン、ヘキサメチルジシラザン、各種オルガノシラン等で表面処理されたものであってもよい。

本発明の補強性シリカ微粉末の比表面積は、50～350m<sup>2</sup>/g、特に80～250m<sup>2</sup>/gであることが好ましい。なお、比表面積は、例えばBET吸着法により求めるこ

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とができる。

【0025】

これら補強性シリカの配合量は、(A)成分100質量部に対し、10質量部以下(即ち、0~10質量部)、好ましくは0質量部以上8質量部以下、より好ましくは0質量部以上5質量部以下である。10質量部を超える量では、硬化物の圧縮永久歪が悪くなってしまう。

【0026】

(F)成分の水の配合量は、(A)成分100質量部に対し、30~200質量部、好ましくは40~120質量部である。30質量部未満では熱伝導性の低下が不十分で、200質量部を超える量では、微細で均一なセルを作ることが難しくなる。10

【0027】

その他の成分として、必要に応じて、石英粉、珪藻土、炭酸カルシウムのような充填剤、補強剤となるシリコン系のレジン、カーボンブラック、導電性亜鉛華、金属粉等の導電剤、窒素含有化合物やアセチレン化合物、リン化合物、ニトリル化合物、カルボキシレート、錫化合物、水銀化合物、硫黄化合物等のヒドロシリル化反応制御剤、酸化鉄、酸化セリウムのような耐熱剤、ジメチルシリコンオイル等の内部離型剤、接着性付与剤、チクソ性付与剤等を本発明の目的を損なわない範囲で配合することは任意とされる。

【0028】

本発明の定着ローラは、芯金に上記シリコンゴム組成物の高熱伝導性硬化物層を形成するものであるが、この場合、芯金の材質、寸法等はロールの種類に応じて適宜選定し得る。20

【0029】

また、シリコンゴム組成物の成形、硬化法も適宜選定し得、例えば注入成形、移送成形、射出成形、コーティング等の方法によって成形でき、加熱により硬化される。この際、ゴムを硬化させる温度条件は、100℃未満、好ましくは80℃以上100℃未満、通常、80~98℃、より好ましくは80~95℃であり、硬化時間は、5分から12時間、好ましくは10分から6時間、より好ましくは15分から2時間である。硬化温度が100℃以上になると、水が揮発してしまい、所望のスポンジが得られなくなってしまう。水を揮発させるための2次キュアとしては、温度条件は、100℃以上、好ましくは150℃以上250℃以下、より好ましくは180℃以上250℃以下で、2次キュア時間は、10分から48時間、好ましくは30分から24時間である。30

【0030】

また、シリコンゴム層の外周に更にフッ素樹脂層やフッ素ゴム層を設けてもよい。この場合、フッ素系樹脂層は、フッ素系樹脂コーティング材やフッ素系樹脂チューブなどにより形成され、上記シリコンゴム層を被覆する。ここでフッ素系樹脂コーティング材としては、例えばポリテトラフルオロエチレン樹脂(PTFE)のラテックスや、ダイエルラテックス(ダイキン工業社製、フッ素系ラテックス)等が挙げられ、またフッ素系樹脂チューブとしては、市販品を使用し得、例えばポリテトラフルオロエチレン樹脂(PTFE)、テトラフルオロエチレン-パーフルオロアルキルビニルエーテル共重合体樹脂(PFA)、フッ化エチレン-ポリプロピレン共重合体樹脂(FEP)、ポリフッ化ビニリデン樹脂(PVDF)、ポリフッ化ビニル樹脂などが挙げられるが、これらのうちで特にPFAが好ましい。40

【0031】

なお、このシリコンゴムスポンジ形成性組成物の硬化物層は、熱伝導率が0.15W/m・℃以下、特に0.05~0.14W/m・℃であることが好ましい。熱伝導率が小さすぎると、ゴム物性が不十分でロール耐久性等に劣ったり、高速プリンターとしての性能が不十分になってしまう場合がある。

【0032】

なお、上記シリコンゴム層の厚さは適宜選定されるが、0.05~80mm、特に0.1~50mmであることが、シリコンゴムのゴム弾性をいかす点で好ましい。また、50

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その上に形成されるフッ素樹脂又はフッ素ゴム層の厚さは、5～200 $\mu$ m、特に10～100 $\mu$ mが好ましい。

## 【実施例】

## 【0033】

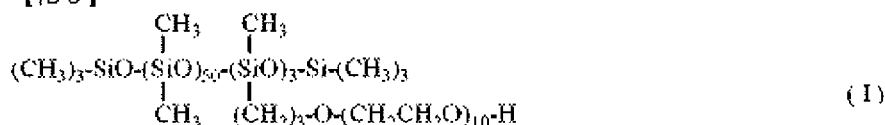
以下、実施例及び比較例を示し、本発明を具体的に説明するが、本発明は下記の実施例に制限されるものではない。なお、下記の例において部及び%はそれぞれ質量部と質量%を示す。また、分子量はGPCによるポリスチレン換算の重量平均分子量である。

## 【0034】

## 【実施例1】

両末端がジメチルビニルシロキシ基で封鎖されたジメチルポリシロキサン（重合度220、ビニル価0.000125mol/g）100部、比表面積が110m<sup>2</sup>/gである疎水化処理されたヒュームドシリカ（日本エアロジル社製、R-972）3部、下記式（I）

## 【化3】



で示されるポリエーテル変性シリコン（分子量5540、分子中のシロキサン量73%）8部をプラネタリーミキサーに入れ、10分撹拌を続けた後、架橋剤として両末端及び側鎖にSi-H基を有するメチルヒドロジェンポリシロキサン（重合度18、Si-H量0.0038mol/g）4.3部（珪素原子結合水素原子/アルケニル基（モル比）=1.3）を添加し、10分撹拌を行った。これに水80部を4回に分けて添加し、トータル1時間撹拌を続けた。最後に反応制御剤としてエチルシクロヘキサノール0.05部を添加し、15分撹拌を続けてできあがった組成物をシリコンゴム組成物（1）とした。

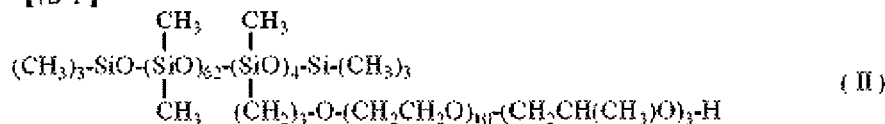
このシリコンゴム組成物（1）に白金触媒（Pt濃度1%）0.1部を混合し、型に流し込み、95℃のオープンに1時間放置して、硬化させた後、型からはずし、200℃で4時間ポストキュアして得た硬化物について、JIS K 6249に基づき、比重、硬さ、圧縮永久歪を測定した結果について表1に記した。また、熱伝導率を熱伝導計（QTM-3 京都電子社製）で測定した結果、及び電子顕微鏡にて断面のセル状態を観察した結果について、同じく表1に記した。

## 【0035】

## 【実施例2】

側鎖ビニル基含有ジメチルポリシロキサン（重合度400、ビニル価0.000094mol/g）50部、両末端がジメチルビニルシロキシ基で封鎖されたジメチルポリシロキサン（重合度180）50部、比表面積が190m<sup>2</sup>/gであるヒュームドシリカ（日本エアロジル社製、アエロジル200）0.5部、下記式（II）

## 【化4】



で示されるポリエーテル変性シリコン（分子量5460、分子中のシロキサン量51%）3部、架橋剤として実施例1のメチルヒドロジェンポリシロキサンを3.2部（珪素原子結合水素原子/アルケニル基（モル比）=1.0）をプラネタリーミキサーに入れ、15分撹拌を行った。その後、水60部を3回に分けて添加し、トータル1時間撹拌を続けた。最後に、反応制御剤としてエチルシクロヘキサノール0.05部を添加し、15分撹拌を続けてできあがった組成物をシリコンゴム組成物（2）とした。

このシリコンゴム組成物（2）に白金触媒（Pt濃度1%）0.1部を混合し、実施

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例 1 と同様に、硬化させ、比重、硬さ、圧縮永久歪、熱伝導率及び断面のセル状態を測定し、表 1 に記した。

【0036】

【実施例 3】

側鎖ビニル基含有ジメチルポリシロキサン（重合度 400、ビニル価 0.000094 mol/g）50 部、両末端がジメチルビニルシロキシ基で封鎖されたジメチルポリシロキサン（重合度 180）50 部、比表面積が  $190 \text{ m}^2/\text{g}$  であるヒュームドシリカ（日本エアロジル社製、アエロジル 200）0.5 部、上記式 (I) で示されるポリエーテル変性シリコン 3 部、架橋剤として実施例 1 のメチルヒドロジェンポリシロキサン 7.0 部（珪素原子結合水素原子/アルケニル基（モル比）= 2.2）をプラネタリーミキサーに入れ、15 分撹拌を行った。その後、水 60 部を 3 回に分けて添加し、トータル 1 時間撹拌を続けた。最後に、反応制御剤としてエチルシクロヘキサノール 0.05 部を添加し、15 分撹拌を続けてできあがった組成物をシリコンゴム組成物 (3) とした。

このシリコンゴム組成物 (3) に白金触媒（Pt 濃度 1%）0.1 部を混合し、実施例 1 と同様に、硬化させ、比重、硬さ、圧縮永久歪、熱伝導率及び断面のセル状態を測定し、表 1 に記した。

【0037】

【実施例 4】

両末端がジメチルビニルシロキシ基で封鎖され、側鎖にビニル基を含有するジメチルポリシロキサン（重合度 550、ビニル価 0.000120 mol/g）100 部、比表面積が  $110 \text{ m}^2/\text{g}$  である疎水化処理されたヒュームドシリカ（日本エアロジル社製、R-972）3 部、上記式 (I) で示されるポリエーテル変性シリコン 8 部をプラネタリーミキサーに入れ、10 分撹拌を続けた後、実施例 1 のメチルヒドロジェンポリシロキサン 4.1 部（珪素原子結合水素原子/アルケニル基（モル比）= 1.3）を添加し、10 分撹拌を行った。これに水 80 部を 4 回に分けて添加し、トータル 1 時間撹拌を続けた。最後に反応制御剤としてエチルシクロヘキサノール 0.05 部を添加し、15 分撹拌を続けてできあがった組成物をシリコンゴム組成物 (4) とした。

このシリコンゴム組成物 (4) に白金触媒（Pt 濃度 1%）0.1 部を混合し、実施例 1 と同様に、硬化させ、比重、硬さ、圧縮永久歪、熱伝導率及び断面のセル状態を測定し、表 1 に記した。

【0038】

【実施例 5】

両末端がジメチルビニルシロキシ基で封鎖された実施例 1 のジメチルポリシロキサン（重合度 220、ビニル価 0.0125 mol/100 g）100 部、比表面積が  $110 \text{ m}^2/\text{g}$  である疎水化処理されたヒュームドシリカ（日本エアロジル社製、R-972）3 部、下記式 (II) で示される非イオン系界面活性剤 8 部をプラネタリーミキサーに入れ、10 分撹拌を続けた後、架橋剤として実施例 1 のメチルヒドロジェンポリシロキサン 4.3 部を添加し、10 分撹拌を行った。これに水 80 部を 4 回に分けて添加し、トータル 1 時間撹拌を続けた。最後に反応制御剤としてエチルシクロヘキサノール 0.05 部を添加し、15 分撹拌を続けてできあがった組成物をシリコンゴム組成物 (5) とした。

【化 5】



(III)

で示される非イオン系界面活性剤 8 部をプラネタリーミキサーに入れ、10 分撹拌を続けた後、架橋剤として実施例 1 のメチルヒドロジェンポリシロキサン 4.3 部を添加し、10 分撹拌を行った。これに水 80 部を 4 回に分けて添加し、トータル 1 時間撹拌を続けた。最後に反応制御剤としてエチルシクロヘキサノール 0.05 部を添加し、15 分撹拌を続けてできあがった組成物をシリコンゴム組成物 (5) とした。

このシリコンゴム組成物 (5) に白金触媒（Pt 濃度 1%）0.1 部を混合し、実施例 1 と同様に、硬化させ、比重、硬さ、圧縮永久歪、熱伝導率及び断面のセル状態を測定し、表 1 に記した。

【0039】

【比較例 1】

実施例 1 の両末端がジメチルビニルシロキシ基で封鎖されたジメチルポリシロキサン（

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重合度 220、ビニル価 0.0125 mol/100 g) 100 部、比表面積が 110 m<sup>2</sup>/g である疎水化処理されたヒュームドシリカ (日本エアロジル社製、R-972) 3 部、上記式 (I) で示されるポリエーテル変性シリコン 8 部をブラネタリーミキサーに入れ、10 分攪拌を続けた後、架橋剤として実施例 1 のメチルヒドロジェンポリシロキサン 4.3 部を添加し、10 分攪拌を行った。最後に反応制御剤としてエチニルシクロヘキサノール 0.05 部を添加し、15 分攪拌を続けてできあがった組成物をシリコンゴム組成物 (6) とした。

このシリコンゴム組成物 (6) に白金触媒 (Pt 濃度 1%) 0.1 部を混合し、実施例 1 と同様に、硬化させ、比重、硬さ、圧縮永久歪、熱伝導率及び断面のセル状態を測定し、表 1 に記した。

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【0040】

【比較例 2】

側鎖ビニル基含有ジメチルポリシロキサン (重合度 400、ビニル価 0.000094 mol/g) 50 部、両末端がジメチルビニルシロキシ基で封鎖されたジメチルポリシロキサン (重合度 180) 50 部、比表面積が 190 m<sup>2</sup>/g であるヒュームドシリカ (日本エアロジル社製、アエロジル 200) 12 部、上記式 (I I) で示されるポリエーテル変性シリコン 3 部、架橋剤として実施例 1 のメチルヒドロジェンポリシロキサン 7.0 部 (珪素原子結合水素原子/アルケニル基 (モル比) = 2.2) をブラネタリーミキサーに入れ、15 分攪拌を行った。その後、水 60 部を 3 回に分けて添加し、トータル 1 時間攪拌を続けた。最後に、反応制御剤としてエチニルシクロヘキサノール 0.05 部を添

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加し、15 分攪拌を続けてできあがった組成物をシリコンゴム組成物 (7) とした。このシリコンゴム組成物 (7) に白金触媒 (Pt 濃度 1%) 0.1 部を混合し、実施例 1 と同様に、硬化させ、比重、硬さ、圧縮永久歪、熱伝導率及び断面のセル状態を測定し、表 1 に記した。

【0041】

【表 1】

	実施例 1	実施例 2	実施例 3	実施例4	実施例5	比較例 1	比較例2
比重 23℃	0.61	0.72	0.74	0.65	0.63	1.01	0.65
硬度 (デュロメータ-A)	12	17	23	15	16	28	27
圧縮永久歪 (%) [150℃×22時間、25%圧縮]	17	13	29	24	25	7	62
熱伝導率 (W/m・℃)	0.10	0.13	0.13	0.11	0.11	0.19	0.11
セル状態	約5μmの 均一なセル	約5μmの 均一なセル	約5μmの 均一なセル	約10μmのセル であるが、 30μm以上の ものも多数	約10μmの 均一なセル	発泡しない ため セルが存在 しない	約10μmのセル であるが、 50μm以上の ものも多数

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【0042】

【実施例 6】

直径 50 mm×長さ 30 mm のアルミニウムシャフトの表面に、付加反応型液状シリコンゴム用プライマー No. 101A/B (信越化学工業社製) を塗付した。このアルミニウムシャフトを設置した金型内に、実施例 1 のシリコンゴム組成物 (1) に触媒を加えたものを充填し、95℃で 1 時間加熱硬化し、更に 180℃で 4 時間ポストキュアし、外径 26 mm×長さ 250 mm のシリコンゴムローラを得た。このシリコンゴムロー

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ラの表面を研磨し、内面に接着剤を塗布した厚さ $50\mu\text{m}$ のPFAチューブをローラ表面に被せ、 $180^\circ\text{C}$ で2時間硬化し、PFA樹脂被覆シリコンゴムローラを作製した。

このローラを $180^\circ\text{C}$ のオープンから取り出し、室温で2分間放置した時のローラ表面温度は $72^\circ\text{C}$ であった。またPPC複写機の定着ローラとして組み込み、通常運転の後、ヒーターローラのヒーターを止めて50枚の通紙が可能であった。

【0043】

【比較例3】

直径 $50\text{mm}$ ×長さ $30\text{mm}$ のアルミニウムシャフトの表面に、付加反応型液状シリコンゴム用プライマーNo. 101A/B（信越化学工業社製）を塗付した。このアルミニウムシャフトを設置した金型内に、比較例1のシリコンゴム組成物（6）に触媒を加えたものを充填し、 $95^\circ\text{C}$ で1時間加熱硬化し、更に $180^\circ\text{C}$ で4時間ポストキュアし、外径 $26\text{mm}$ ×長さ $250\text{mm}$ のシリコンゴムローラを得た。このシリコンゴムローラの表面を研磨し、内面に接着剤を塗布した厚さ $50\mu\text{m}$ のPFAチューブをローラ表面に被せ、 $180^\circ\text{C}$ で2時間硬化し、PFA樹脂被覆シリコンゴムローラを作製した。

このローラを $180^\circ\text{C}$ のオープンから取り出し、室温で2分間放置した時のローラ表面温度は $46^\circ\text{C}$ であった。またPPC複写機の定着ローラとして組み込み、通常運転の後、ヒーターローラのヒーターを止めて通紙を続けたところ、25枚目より定着不十分で滲みが発生し始めた。

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